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HOMOGENEOUS AND HETEROGENEOUS RUBBERY/RUBBERY DIBLOCK COPOLYMER--ETC(U)  
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Technical Report No. 8

HOMOGENEOUS AND HETEROGENEOUS RUBBERY/RUBBERY DIBLOCK COPOLYMERS  
AND POLYMER BLENDS - A UNIFIED VIEW.

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18. SUPPLEMENTARY NOTES Based on a lecture to be presented at the August 1981 meeting of the Polymer Division of the American Chemical Society, Symposium on Elastomers and Rubber Elasticity.			
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) block copolymers, polymer blends, morphology, homogeneity in polymer mixtures, microphase separation			
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A large amount of data obtained in previous work is brought together to present a unified picture of the homogeneous/heterogeneous behavior of various two-component polymer systems. The materials under discussion are comprised of various proportions of a rubbery/rubbery diblock copolymer and each of the corresponding rubbery homopolymers.			

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Introduction. For the past several years, research in our laboratory has been directed at obtaining an understanding of the influence of molecular architecture and of bulk morphology on the physical properties of: (i) diblock copolymers, (ii) binary blends of the corresponding homopolymers, and (iii) binary and ternary blends of a diblock copolymer with either or both of the corresponding homopolymers. In many cases the polymeric components were selected so that both exhibited rubbery behavior ( $T > T_g$ ) at room temperature (1-7). Reasons for the selection of rubbery/rubbery systems included the following: Relatively little attention has been given to rubbery/rubbery diblock copolymers in spite of the fact that there are numerous applications for blends of rubbery components. Furthermore, it is likely that having both components of our block copolymers and polymer blends in the amorphous rubbery state will help to minimize nonequilibrium effects which strongly influence the structure (8,9) and properties (10) of glassy/glassy or, glassy/rubbery systems. Finally, rubbery/rubbery systems based on diene polymers offer opportunities for investigation of changes in molecular architecture in the absence of changes in chemical composition. The purpose of the present paper is to provide a unified framework in which to view the previous results and to provide guidance regarding the formulation of rubbery/rubbery compositions with specified properties.

Results. A summary of some of the results of our investigations are presented in Tables 1 and 2. The assessment of the homogeneity or heterogeneity of a particular material is always based on the combined information from three types of experiments: (i) thermal analysis, (ii) dynamic mechanical testing and (iii) transmission electron microscopy. Other researchers have



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also pointed out the need for applying a variety of techniques in the examination of homogeneous and heterogeneous polymer blends (11). Table 1 describes results obtained on samples of either pure diblock copolymer or simple binary blends of homopolymers. Table 2 on the other hand presents data obtained for binary and ternary blends of certain diblock copolymers (1,4 polybutadiene-b-cis 1,4 polyisoprene) with either or both of the corresponding homopolymers. Previously (2,5), ternary composition diagrams based on the weight percentage of each of the three components (diblock/1,4 polybutadiene homopolymer/cis 1,4 polyisoprene homopolymer) were used to summarize results and to analyze trends in the data. For reasons to be described below, the composition data provided in Table 2, namely the mole fraction of polybutadiene repeat units in a blend and the weight fraction of diblock in that blend, will be useful for obtaining a clearer understanding of the behavior of these binary and ternary polymer mixtures.

Discussion. Questions arise in considering whether or not the vast array of data in Tables 1 and 2 can be explained in a concise and unified way. For example is it reasonable that homopolymer blends of 1,4B and I are heterogeneous whereas the corresponding 1,4B-b-I diblocks are homogeneous? Likewise why are homopolymer blends of 1,4B and 1,2B, although uniform in chemical composition ( $C_4H_6$ ), heterogeneous in all proportions; the corresponding 1,4B-b-1, 2B diblocks are heterogeneous at low molecular weights and nearly equal block size, and become homogeneous as molecular weight and block size disparity increase. Why do high vinyl polybutadiene (vinyl B) and pure 1,2 polybutadiene (1,2B) both form homogeneous mixtures with cis 1,4 polyisoprene whereas all of the other polybutadiene

microstructures (1,4B; cis B; trans B) form heterogeneous blends with cis 1,4 polyisoprene? Finally it is interesting that certain binary and ternary mixtures (1,4B + I + 1,4B-b-I diblock) are homogeneous and some are heterogeneous.

An understanding of some of the points mentioned above can be obtained by considering the various theories (14,15,16) which have been developed to describe microphase separation in block copolymers. Meier (14) pointed out that as molecular weight is increased, homopolymers begin to form heterogeneous mixtures earlier than the corresponding diblock copolymer; an estimate of 2.5 to 5 was given for the ratio [critical molecular weight for diblock domain formation]/[critical molecular weight for phase separation in homopolymer blends]. More recent work by Helfand (15) supports this conclusion. Very recently Leibler (16) suggested that the critical molecular weight ratio should be  $10.5/2.0 = 5.25$  under the reasonable assumption that the segmental interaction parameter  $\chi$  is the same in the diblock and the blend. With these ideas in mind, Figure 1 provides a unified view of much of the data in Table 1. Certain samples, particularly the 1,4B-b-I diblocks and the blends of 1,4B and I, fall in the region in which homogeneous block copolymer and heterogeneous blends are expected, thus accounting for the earlier observations (Table 1, 2-5). Also in agreement with Table 1, the 1,4B-b-1,2B diblocks closely follow the boundary between the regions of homogeneous and heterogeneous blend behavior. Data for blends of 1,2B and I and of vinyl B and I fall below the lower curve, i.e. in the region of homogeneity for both block copolymers and polymer blends: the corresponding diblocks are thus expected to be homogeneous but have not yet been synthesized. Finally some points have been added to the graph to indicate the location of representative polystyrene/polybutadiene block

copolymers which have received considerable attention in the literature.

An explanation of the behavior of the binary and ternary blends of Table 2 requires a three dimensional representation as shown in Figure 2. The curve in the plane at the right side is the boundary between homogeneous and heterogeneous diblocks (i.e. the upper curve in Figure 1) while the left-most plane contains a similar boundary for pure homopolymer blends (lower curve in Figure 1). Clearly the location of the boundaries on intermediate planes, representing various weight percentages of diblock copolymer (column 3 of Table 2), must vary smoothly between the position on the right and on the left. At present a linear variation of this position with diblock weight percentage has been assumed. In this way, the behavior of many of the blends in Table 3 can be explained by examining the planes at 0.25, 0.50 and 0.75 weight fraction of diblock copolymer. These plots are shown in Figure 3. A more sophisticated method of representing these plots, using three dimensional projections, has been worked out by Bates (13).

At present, all of the explanations are put forward on a semiquantitative basis. What is needed for an absolute assignment of a vertical location of any point in Figures 1-3 is a known value of  $M_{crit}$  (the critical molecular weight for phase separation of the block copolymer) or alternatively a  $\chi_{AB}$  value for each pair. Also the shapes of the boundaries are not necessarily invariant with the choice of various A-B pairs or with the location of the plane in Figure 2. Ongoing work is aimed at providing reasonable estimates for the various values of  $M_{crit}$ . We are also in the process of using existing theories (15,17) to determine the sensitivity of the shape and location of the various boundaries to such parameters as the Kuhn segment length (15) and the interaction parameter  $\chi_{AB}$  (17).

Acknowledgements. This work is sponsored by the Office of Naval Research. Data presented in Tables 1 and 2 are taken from thesis research conducted at MIT by the following: Alonso R. Ramos, David E. Wilfong and Jose M. Torradas. Acknowledgement is also made to Frank S. Bates for assistance with the calculations in Table 2 and for helpful discussions regarding Figures 2 and 3.

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Table 1 Summary of Results Obtained on Various Rubbery/Rubbery Diblock Copolymers  
and Homopolymer Blends (1-6)

<u>Component A<sup>(a)</sup></u>	<u>Component B<sup>(a)</sup></u>	<u>Diblock or Blend</u>	<u>Mole Fraction A</u>	<u>Molecular Weight<sup>(b)</sup></u>	<u>Homogeneous?</u>
1,4B	I	Blend	0.296	133000	No
1,4B	I	Blend	0.557	133000	No
1,4B	I	Blend	0.704	133000	No
1,4B	I	Diblock	0.500	250000	Yes
1,4B	I	Diblock	0.660	264000	Yes
1,4B	I	Diblock	0.340	270000	Yes
trans B	I	Blend	0.557	156000	No
cis B	I	Blend	0.557	416000	No
vinyl B	I	Blend	0.557	468000	Yes
1,2B	I	Blend	0.557	150000	Yes
1,4B	vinyl B	Blend	0.500	468000	No
1,4B	1,2B	Blend	0.200	100000	No
1,4B	1,2B	Blend	0.625	100000	No
1,4B	1,2B	Blend	0.880	100000	No
1,4B	1,2B	Diblock	0.625	80000	No
1,4B	1,2B	Diblock	0.769	130000	Yes
1,4B	1,2B	Diblock	0.833	180000	Yes
1,4B	1,2B	Diblock	0.869	230000	Yes

(a) 1,4B = polybutadiene (45% cis 1,4; 45% trans 1,4; 10% 1,2)...I = cis 1,4 polyisoprene...trans B = 6% cis 1,4; 91% trans 1,4; 3% 1,2...cisB = 92% cis 1,4; 4% trans 1,4; 4% 1,2...Vinyl B = 20% cis 1,4; 16% trans 1,4; 64% 1,2...1,2 B = 99% 1,2 polybutadiene (12,1)

(b) For homopolymer blends, the value given is the higher molecular weight; for diblock copolymers overall molecular weight is given.

Table 2 Summary of Results Obtained on Blends of Diblock Copolymers with the Corresponding Homopolymers (2,5)

<u>Type of Blend<sup>(a)</sup></u>	<u>Mole Fraction of 1,4B in Blend<sup>(b)</sup></u>	<u>Wt. Fraction of Diblock in Blend<sup>(c)</sup></u>	<u>Homogeneous?</u>
Binary: 1,4B + Diblock 3	0.836	0.250	Yes
Binary: 1,4B + Diblock 4	0.921	0.250	Yes
Binary: 1,4B + Diblock 8	0.853	0.250	No
Binary: I + Diblock 3	0.135	0.250	Yes
Binary: I + Diblock 4	0.184	0.250	No
Binary: I + Diblock 8	0.089	0.250	Yes
Ternary: 1,4B + I + Diblock 3	0.415	0.250	No
Ternary: 1,4B + I + Diblock 3	0.664	0.250	No
Ternary: 1,4B + I + Diblock 4	0.702	0.250	No
Ternary: 1,4B + I + Diblock 4	0.459	0.250	No
Ternary: 1,4B + I + Diblock 8	0.394	0.250	No
Ternary: 1,4B + I + Diblock 8	0.627	0.250	No
Binary: 1,4B + Diblock 3	0.764	0.500	Yes
Binary: 1,4B + Diblock 4	0.839	0.500	Yes
Binary: 1,4B + Diblock 8	0.695	0.500	No
Binary: I + Diblock 3	0.262	0.500	Yes
Binary: I + Diblock 4	0.356	0.500	No
Binary: I + Diblock 8	0.175	0.500	Yes
Ternary: 1,4B + I + Diblock 3	0.528	0.500	No
Ternary: 1,4B + I + Diblock 4	0.611	0.500	No
Ternary: 1,4B + I + Diblock 8	0.450	0.500	No
Binary: 1,4B + Diblock 3	0.636	0.750	Yes
Binary: 1,4B + Diblock 4	0.774	0.750	Yes
Binary: 1,4B + Diblock 8	0.525	0.750	No
Binary: I + Diblock 3	0.383	0.750	Yes
Binary: I + Diblock 4	0.516	0.750	No
Binary: I + Diblock 8	0.259	0.750	Yes
Ternary: 1,4B + I + Diblock 3	0.497	0.050	No
Ternary: 1,4B + I + Diblock 3	0.497	0.100	No
Ternary: 1,4B + I + Diblock 3	0.497	0.200	No

Table 2 Continued

Type of Blend <sup>(a)</sup>	Mole Fraction of 1,4B in Blend <sup>(b)</sup>	Wt. Fraction of Diblock in Blend <sup>(c)</sup>	Homogeneous?
Ternary: 1,4B + I + Diblock 3	0.546	0.333	No
Ternary: 1,4B + I + Diblock 3	0.497	0.400	No
Ternary: 1,4B + I + Diblock 3	0.497	0.300	Yes
Ternary: 1,4B + I + Diblock 4	0.598	0.333	No
Ternary: 1,4B + I + Diblock 8	0.339	0.200	No
Ternary: 1,4B + I + Diblock 8	0.498	0.333	No
Ternary: 1,4B + I + Diblock 8	0.339	0.400	No
Ternary: 1,4B + I + Diblock 8	0.339	0.600	No
Ternary: 1,4B + I + Diblock 8	0.339	0.800	No

(a) I = cis 1,4 polyisoprene (M = 133000)...1,4B = polybutadiene (45% cis 1,4; 45% trans 1,4; 10% 1,2) (M = 120000)...Diblock 3 = 1,4B-b-I (110000-b-140000)...  
Diblock 4 = 1,4B-b-I (161000-b-103000)...Diblock 8 = 1,4B-b-I (78000-b-192000):  
 Careful HPSEC analysis of Diblock 8 shows that unlike the other two diblocks a significant fraction (10% by weight) of 1,4B homopolymer is present in this sample. This fact is not taken into account in the above tabulation nor in previous discussions of these data (2). However correcting for the homopolymer content would result in only minor changes above as follows: Column 1 - Diblock 8 must now be described as 1,4B-b-I + 10% 1,4B (78000-b-293000 + 10% of 78000 1,4B):  
 Column 2 - No changes; Column 3 - wt fraction diblock decreases slightly in all cases, e.g. 0.750 becomes 0.675, 0.500 becomes 0.450, 0.250 becomes 0.225;  
 Column 4 - No changes.

(b) Mole fraction of 1,4B includes contribution from 1,4 polybutadiene moiety of the diblock and from the 1,4 polybutadiene homopolymer, if any.

(c)  $[\text{weight of diblock}]/[\text{weight of diblock plus homopolymer(s)}]$

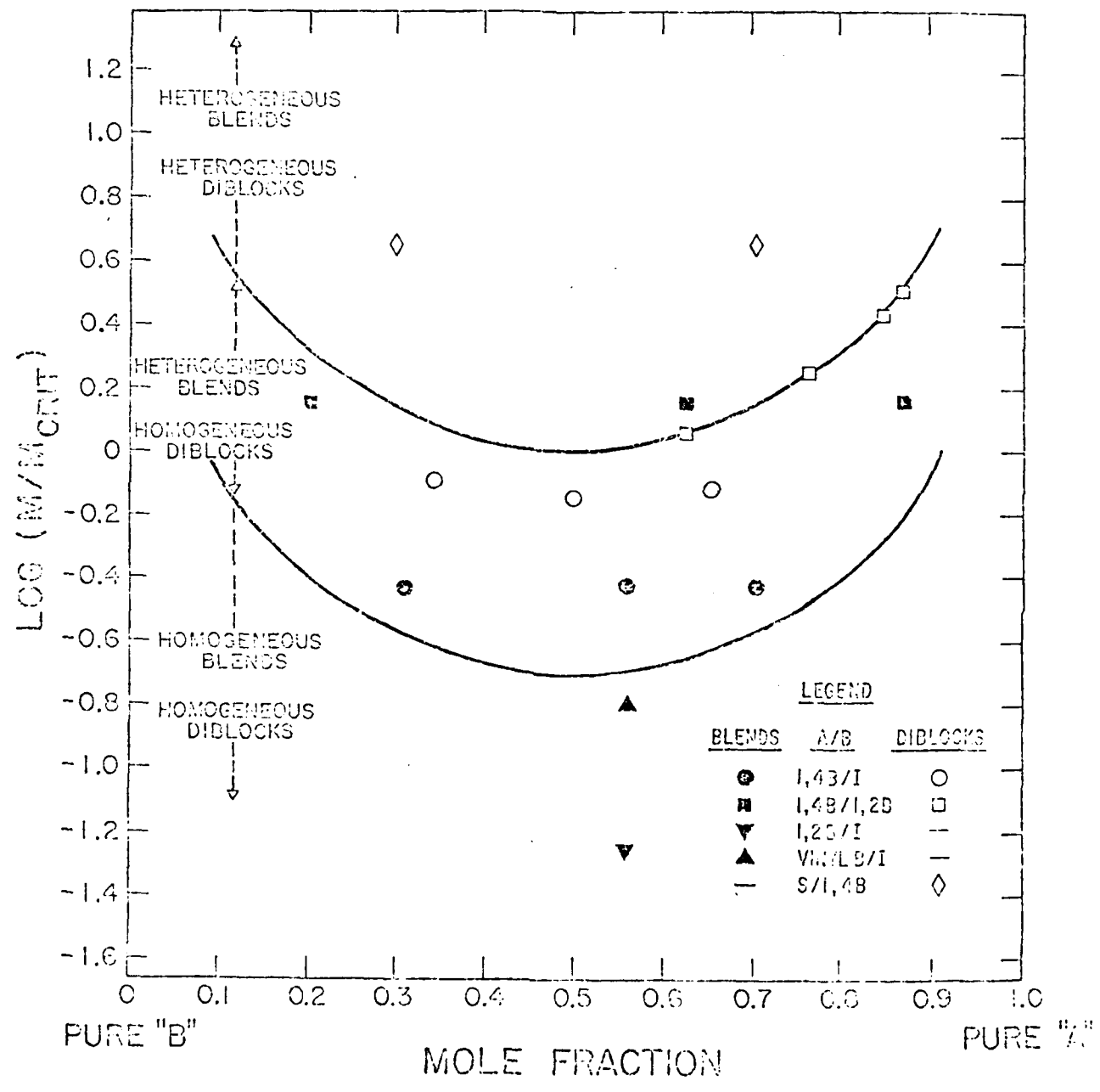
### Figure Legends

Figure 1. Plot of the logarithm of reduced molecular weight against composition for various diblock copolymers and homopolymer blends. Shapes of the curves were calculated based on a modified version of Helfand's Fortran program (15). Values of the critical molecular weight are not precisely known for each AB pair but all data points for a given set are plotted correctly relative to each other. For example, knowing from previous work (2-5) that homopolymer blends of 1,4B and I are heterogeneous places the solid circle points (●) above the lower curve in this figure; with this constraint the question arises whether or not the points (●) for the corresponding diblock copolymers of higher molecular weight fall above (heterogeneous) or below (homogeneous) the upper curve. As seen in the figure, the points fall below the upper curve, consistent with the previous experimental results (2-5) which indicated that these copolymers are homogeneous materials. Similar considerations apply to the relative locations of the 1,2B/1,4B data points (■, □); the fact that one of the diblocks was heterogeneous and the others were homogeneous is consistent with the fact that all the data points for these copolymers fall close to boundary between homogeneous and heterogeneous diblock copolymer behavior.

Figure 2. Schematic representation of a unified picture for explaining the behavior of blends containing various proportions of homopolymer A, homopolymer B and the corresponding A/B diblock copolymer. The plane at the far right is for pure diblock and the minimum in the curve is located at  $\log M/M_{crit} = 0$ . The minimum in the curve in the plane at the far left (homopolymer blend in the absence of diblock) is located at  $\log M/M_{crit} = \log 2/10.5 = -0.720$  (see references 15-17). In the absence of any guiding theory, a linear variation with weight fraction of diblock has been assumed

to describe the location of the minimum of the curve on intermediate planes.

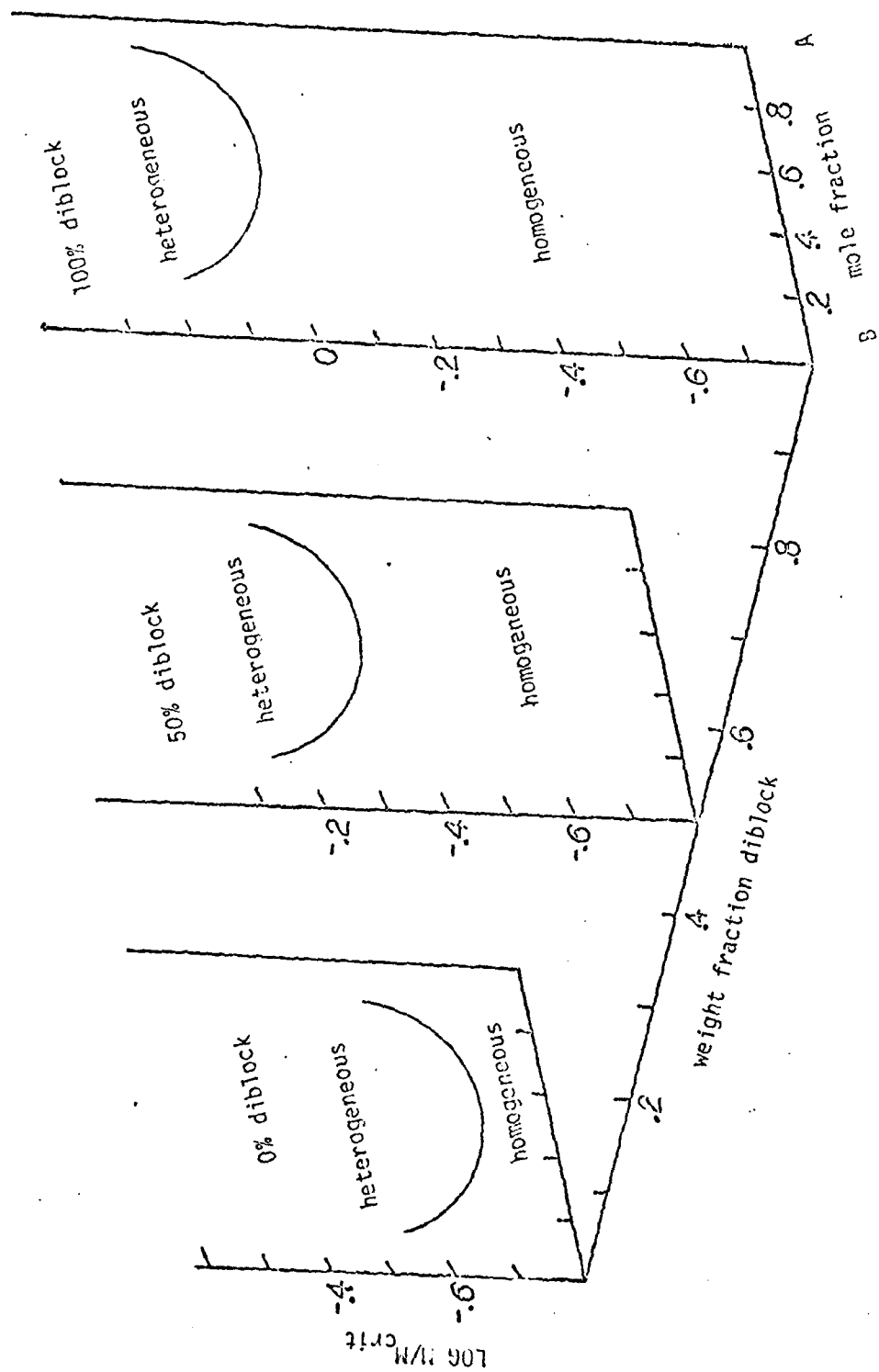
Figure 3. Plots of data from Table 2 for three of the planes of Figure 2 for the system 1,4B homopolymer, I homopolymer, 1,4B-b-I diblock. In all cases the molecular weight of the binary or ternary blend was taken to be that of the highest molecular weight component which was the block copolymer in all cases. Figure 3a contains six data points corresponding to the six different blends containing 75% diblock which were examined in the previous experimental work (2-5). Figures 3b and 3c contain nine and twelve points respectively corresponding to blends at 50% and 25% diblock. The varying number of data points is a consequence of the way in which blends were selected for study in the earlier work in which triangular composition diagrams were employed for guidance in the experiments (5,2). Figure 3d is a reduced composite plot of the three diagrams.



Figure

Figure 2

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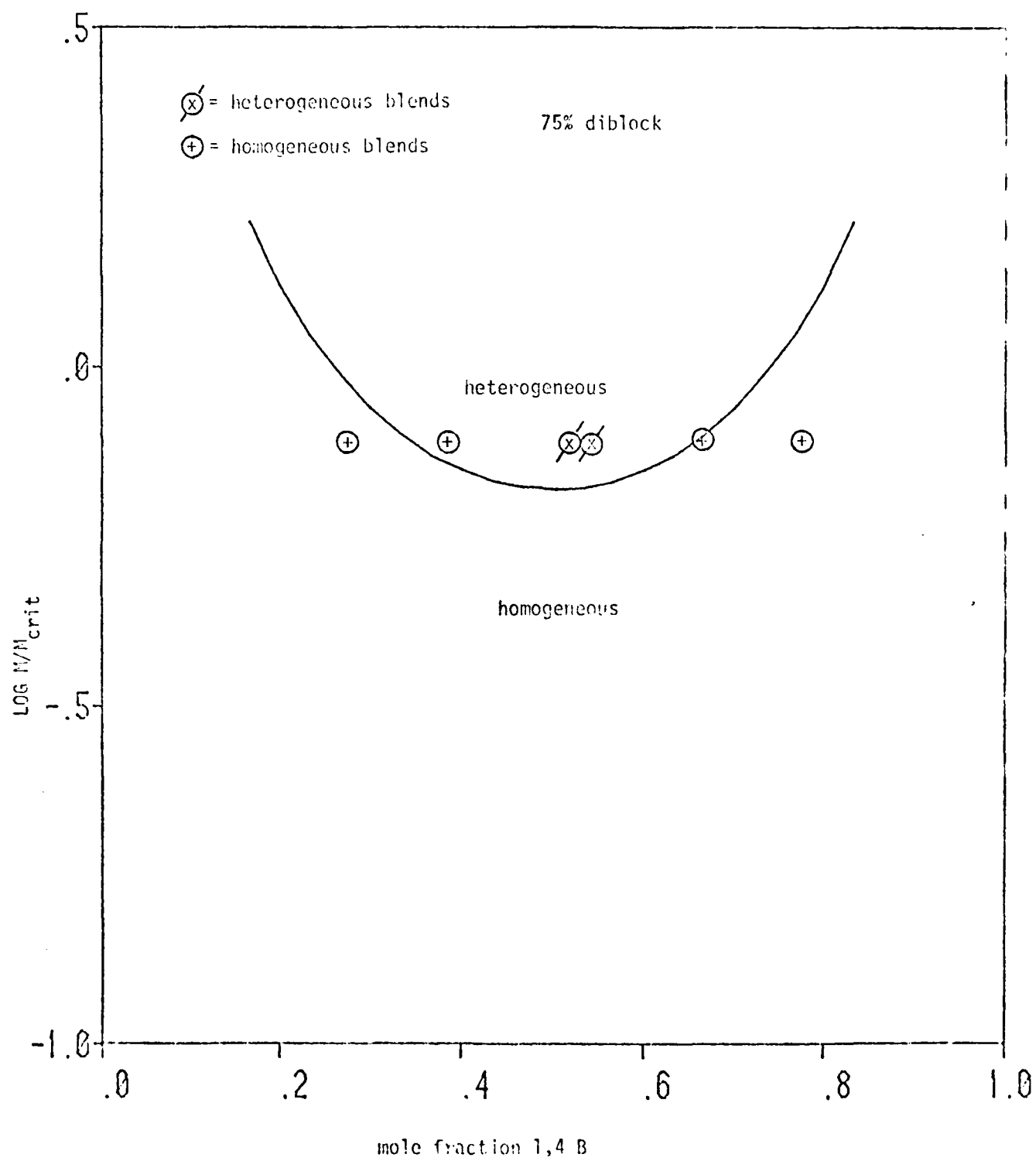


Figure 3a

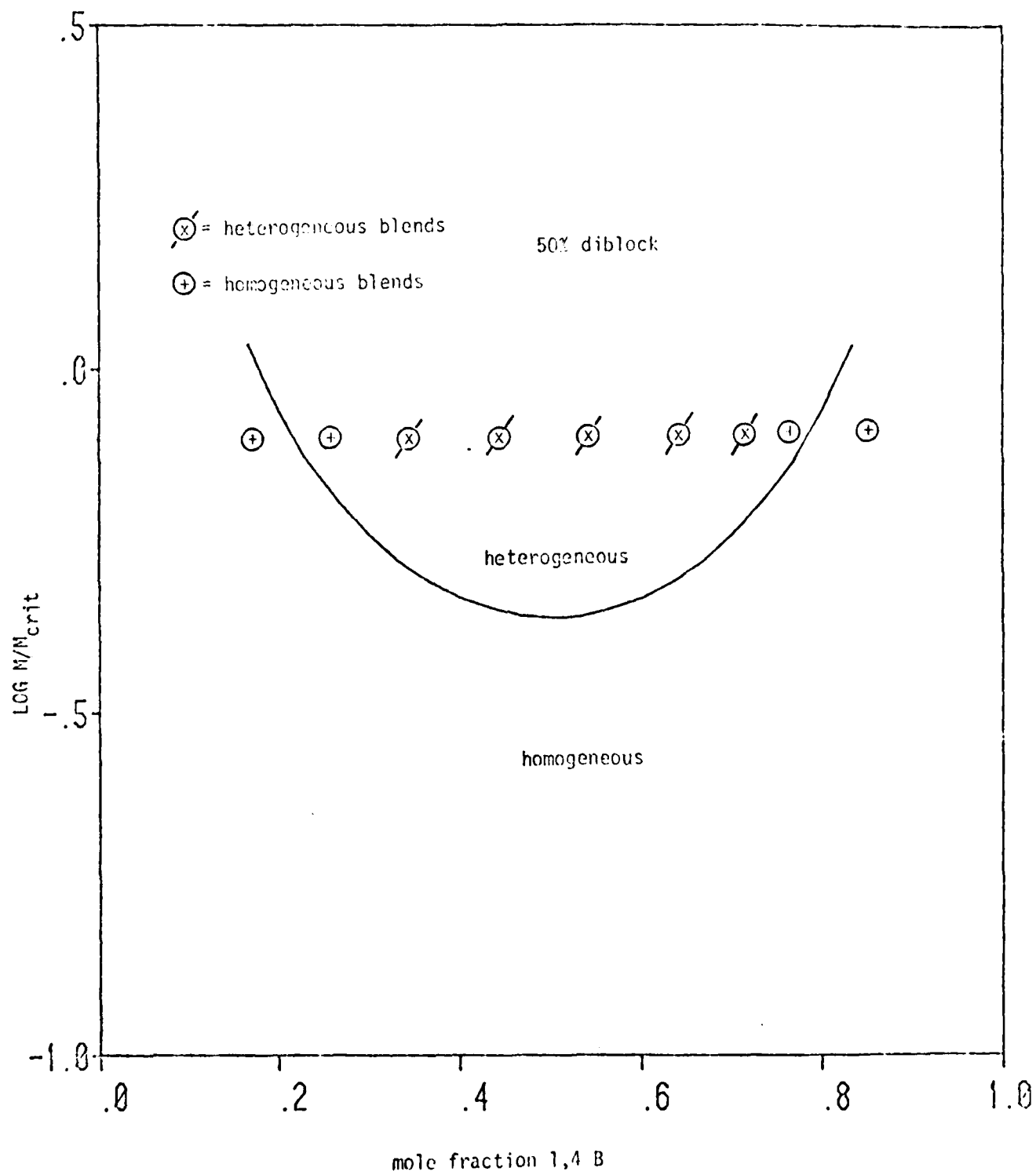


Figure 3b

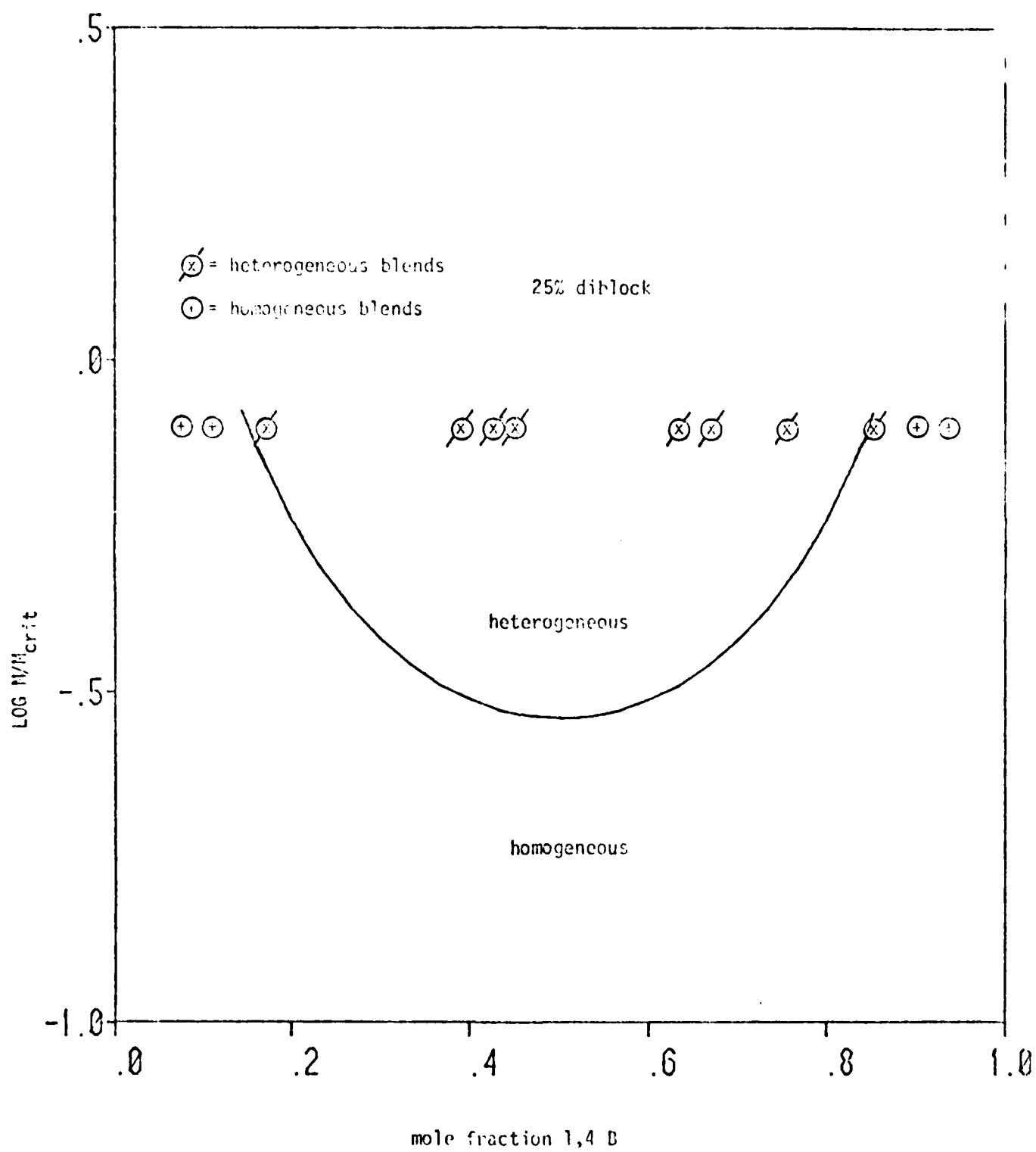


Figure 3c

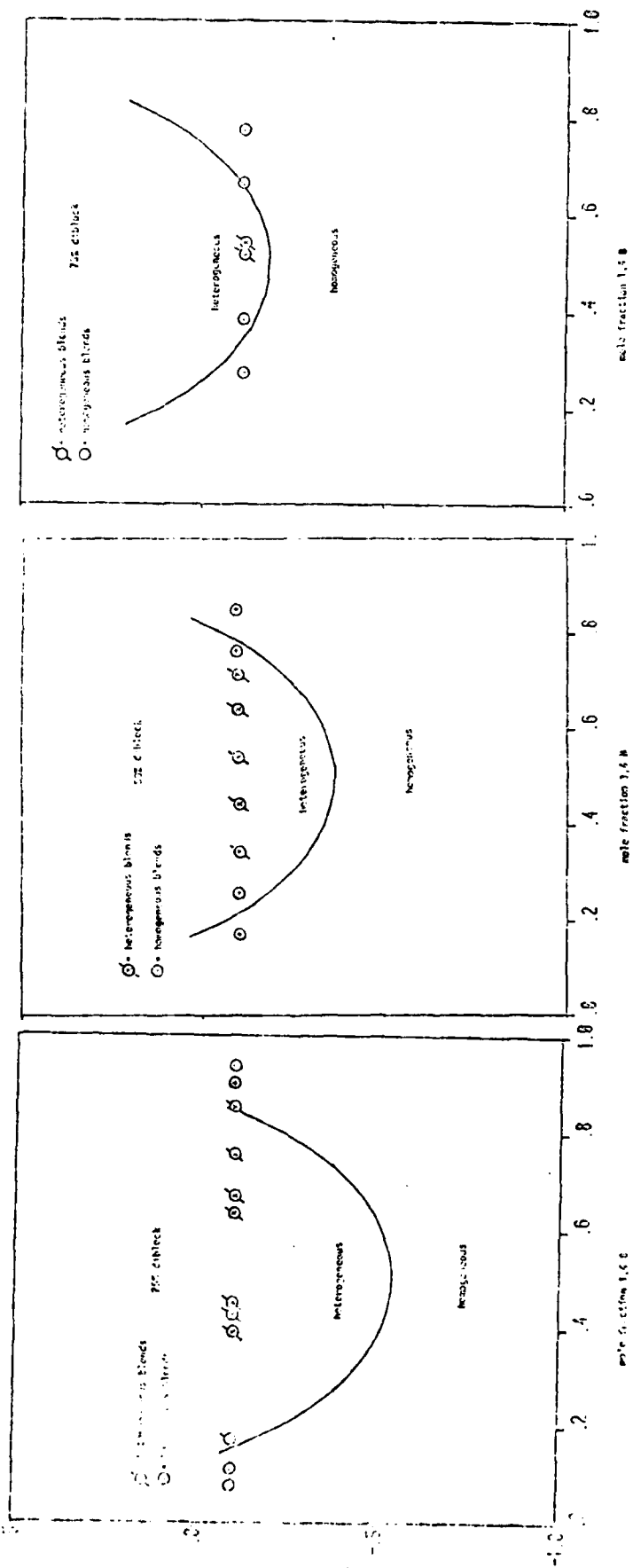


Figure 3d

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